

A comparative study of selected physical properties of five root-canal sealers

F. R. S. McMichen¹, G. Pearson², S. Rahbaran¹ & K. Gulabivala¹

¹Department of Conservative Dentistry, Eastman Dental Institute for Oral Health Care Sciences, University College London, and

²Department of Biomaterials related to Dentistry, Queen Mary University of London, London, UK

Abstract

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Aim To investigate selected physical properties of five root-canal sealers.

Methodology The solubility, film thickness, flow, working and setting times of Roth 801[®], Tubli-Seal EWT[®], AH Plus[®], Apexit[®] and Endion[®] were evaluated. Solubility was measured by weight change of standard specimens in water over a 3-month period. Film thickness was measured as the distance between weighted glass slides containing a standard quantity of sealer. Flow was assessed by rate of sealer extrusion through a standard bore diameter, after unit time. Working time was taken as the point at which flow rate was reduced by 10%. The indentation test using a modified Gilmore needle was employed to investigate the setting times.

Results AH Plus[®] was the least soluble, whilst Apexit[®] was the most unstable in water. The solubility values in increasing order were: AH Plus[®] < Tubli-Seal

EWT[®] < Endion[®] < Apexit[®]. Also, AH Plus[®] had the highest film thickness. All sealers showed comparable flow rates. The working time varied from 50 min for Endion[®] to over 2 h for AH Plus[®] and Tubli-Seal EWT[®]. The range of setting times recorded was from 70 min for Tubli-Seal EWT[®] to 8 days for Roth 801[®].

Conclusions

- 1 AH Plus[®] showed the greatest stability in solution and Tubli-Seal EWT[®] performed well, but Apexit[®] and Endion[®] had higher solubility values.
- 2 The film thickness values in increasing order were: Tubli-Seal EWT[®] < Apexit[®] < Endion[®] = Roth 801[®] < AH Plus[®].
- 3 The flow rates for all sealers were similar.
- 4 The working times for all sealers were greater than 50 min.
- 5 Roth 801[®] did not set when incubated in volumes sufficient to fill the test matrices.

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Introduction

The sealer performs several functions during the obturation of a root-canal system with gutta-percha: It lubricates and aids the seating of the master gutta-percha cone, acts as a binding agent between the gutta-percha and the canal wall and fills anatomical spaces the primary filling material has failed to reach. Root-canal sealers, although used only as adjunctive materials in

the obturation of root-canal systems, have been shown to influence the outcome of root-canal treatment (Ørstavik *et al.* 1987, Eriksen *et al.* 1988). Several studies have evaluated periradicular repair histologically in monkey (Tagger & Tagger 1989) and dog (Leonardo *et al.* 1997, Tanomaru Filho *et al.* 1998) experimental models after root-canal filling with different sealers. Calcium hydroxide-containing sealers have been found to induce biological 'sealing' with calcified tissue deposition over the apical foramina. Any therapeutic effect of this type of sealer is, however, dependent on the calcium hydroxide being in ionized form. This implies that the material must be at least partly soluble.

The handling properties of root-canal sealers and their clinical behaviour may be characterized by

Correspondence: Dr K. Gulabivala, Department of Conservative Dentistry, Eastman Dental Institute for Oral Health Care Sciences, University College London, 256 Grays Inn Road, London WC1X 8LD, UK (Tel.: +44 207 915 1033/1027; fax: +44 207 915 1028; e-mail: K.Gulabivala@eastman.ucl.ac.uk).

laboratory tests on their physical properties. These include flow, solubility, working and setting times. These materials require adequate working time for clinical handling. Within the root canal, their flow properties determine how effectively accessory canals and voids between master and accessory cones may be obturated.

As sealers in unset form may induce adverse tissue reactions, the setting time should not be excessively long. Film thickness and solubility may also affect the sealing ability of sealers (Georgopoulou *et al.* 1995, Kontakiotis *et al.* 1997).

This study compares selected physical properties of five types of root-canal sealers: zinc oxide–eugenol [ZOE] sealer (Roth 801[®]), modified ZOE sealer (Tubli-Seal EWT[®]), modified epoxy resin sealer (AH Plus[®]), calcium hydroxide-containing sealer (Apexit[®]) and glass ionomer sealer (Endion[®]).

Materials and methods

The sorption and solubility, film thickness, flow, working and setting times of five root-canal sealers were investigated. The sealers were Roth 801[®] (Batch # 1042, Roth International Ltd, Chicago, IL, USA), Tubli-Seal EWT[®] (Batch # 4-1269, Kerr Co., Romulus, MI, USA), AH Plus[®] (Batch # 941229, Dentsply Ltd, Weybridge, UK), Apexit[®] (Batch # 713602, Ivoclar-Vivadent, Leicester, UK) and Endion[®] (Batch # 53162, Voco, Cuxhaven, Germany). All sealers were mixed according to the manufacturer's instructions. Roth 801[®] was mixed in a powder:liquid ratio of 0.13 g : 0.03 g.

Sorption and solubility

Long-term solubility and sorption measurements were carried out over a period of 12 weeks. Fifty copper cylindrical matrices (5 mm diameter, 3 mm height) were prepared and preweighed (± 0.0001 g) on an electronic balance (Precisa 120A, PAG Oerlikon AG, Zurich, Switzerland). Ten samples of each of the five sealers were mixed according to the manufacturer's instructions and packed into the matrices. The materials were then stored for 48 h at 37 °C and 100% relative humidity (RH). At the end of this period, all test specimens were set except for Roth 801[®]. This sealer was excluded from further solubility testing.

All set samples were weighed (W_0) to establish a base-line value. They were then placed in preweighed beakers containing 100 mL of distilled, deionized water and left for a further 2 days at 37 °C. The samples were then removed from the containers, blot-dried on absorbent

paper, re-weighed (W_{11}) and replaced in the containers. This procedure was repeated after 3 days, and thereafter at weekly intervals for a period of 12 weeks. The specimens were then desiccated and weighed at weekly intervals until a constant weight was obtained.

Additionally, the storage solutions were evaporated to dryness, and the residue was weighed.

Film thickness

The sealers were mixed in sufficient quantities to cover 3 cm × 3 cm glass slides. In all cases, considerably more material than that normally mixed was required for the test. The combined thickness of the two glass slides was measured first with a set of digital callipers accurate to 0.01 mm (Digimatic Callipers, Mitutoyo UK Ltd, UK). The sealers were then mixed and transferred onto one of the glass slides. The second glass slide was placed centrally over the first. The assembly was then loaded with a 200-g weight and transferred to an oven at 37 °C and 100% RH for 7 min (10 min after the start of the mix). The procedure was repeated five times for each sealer ($n = 5$).

Flow

Two experiments were conducted to measure this characteristic.

Flow measurement by sealer extrusion through a bore

Disposable 1-mL syringes (Plastipak, Becton Dickinson, Dublin, Ireland) were filled with tap water as a control. The syringe was placed in a stabilizing jig on the platform of a tensile testing machine (Hounsfield H25K, Redhill, UK). The time taken for 1 mL of water to be expelled at a constant speed of 125 cm min⁻¹ was measured. The same procedure was carried out for 0.2 mL of test materials (using a new syringe each time); five readings were taken for each sealer. The time taken to expel 0.2 mL of water was taken as a fifth of the time taken to expel 1.0 mL.

Working time

The working time was tested as a function of sealer flow, using the flow determination by measurement of specimen diameter. Sufficient sealer was mixed to fill a disposable 1-mL syringe. Aliquots of 0.5 mL were extruded onto 6 cm × 6 cm squares of polyvinyl chloride film

(Cling Film[®], J. Sainsbury plc, London, UK). Three minutes later, the first sample was placed on a glass slab, covered with a second piece of cling film and a second glass slab, and a 450-g weight was placed on top. After 10 min, the minimum and maximum diameters of the sample discs were measured. This procedure was repeated for the remaining samples. Measurements were made at 10-min intervals. Diameter variation was taken to represent changes in viscosity of the material. The end of the working time was taken as the point at which the flow was reduced by 10% of the original measurement.

Setting time

Setting time was determined using an indentation test. Five specimens of each material were prepared and stored, as for the solubility tests. At specified time intervals, the samples were tested with a hand-held modified Gilmore needle using light pressure. The setting time was taken as the point at which the needle could no longer indent the surface of the sample. All sealers were tested prior to placement in the humidity chamber and then 30 min after starting to mix. If there was any indication of sealer thickening, tests were carried out at 5-min intervals.

With Roth 801[®], a modification of this test was carried out, as the sealer did not set over the 3-month period. Five samples were mixed on glass slides in the powder:liquid ratio of 0.13 g : 0.03 g. The slides and sealer were placed in sealed containers at 37 °C and 100% RH. The samples were then tested on a daily basis with the modified Gilmore needle.

Results

Sorption

The sorption of the materials, a combination of two effects: uptake and dissolution, is calculated from the formula:

$$\frac{(W_t - W_0)}{W_0} \times 100\%$$

where W_t is the weight of the sample at any given time and W_0 is the starting weight. The changes for all the sealers with respect to time are illustrated in Fig. 1. By the end of week 12, all AH Plus[®] specimens had lost weight. The sorption for AH Plus[®] at 12 weeks was -0.08% (Table 1). Apexit[®] showed progressive weight loss throughout. The specimens were tacky to blot-dry, and staining remained on the tissue. At 12 weeks, the

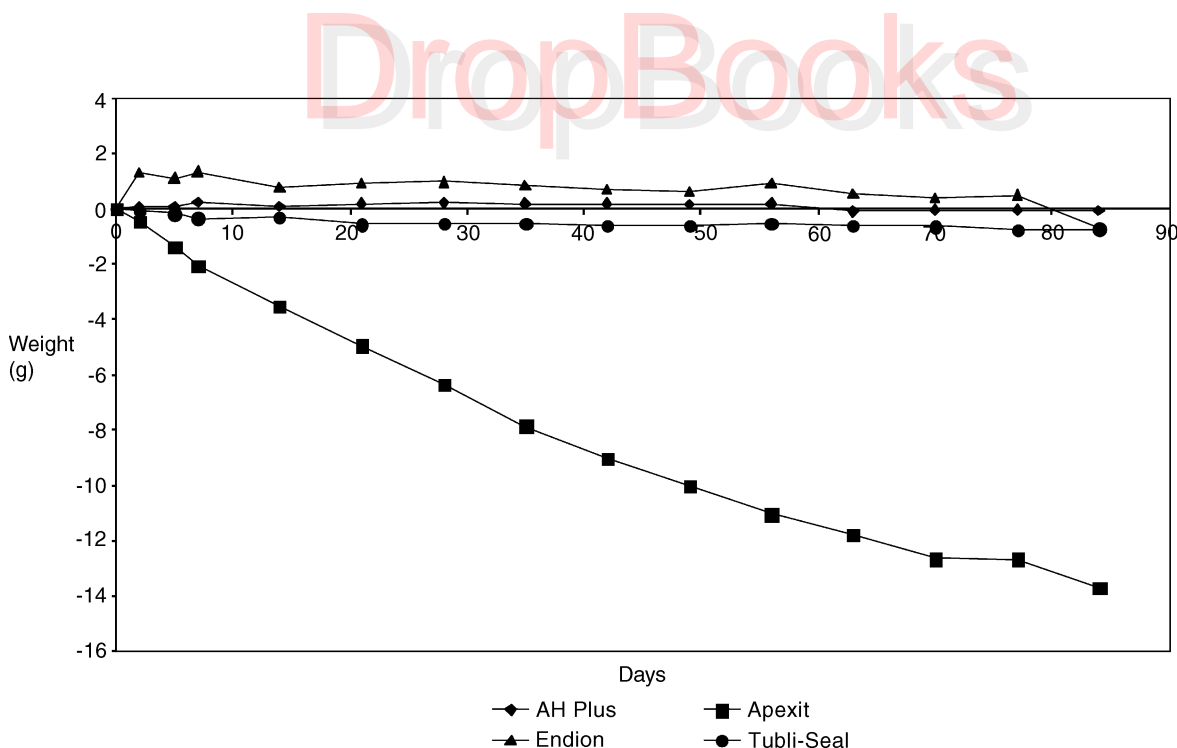


Figure 1 Mean weight changes of the sealers over 12 weeks.

Table 1 Percentage sorption, solubility and residue data

	AH Plus [®] (%)	Apexit [®] (%)	Endion [®] (%)	Tubli-Seal EWT [®] (%)
Sorption ($(W_t - W_0)/W_0 \times 100\%$)	-0.08	-13.75	-0.68	-0.73
Solubility ($(W_0 - W_f)/W_0 \times 100\%$)	0.32	16.43	8.19	1.79
Residue as % of original	0.11	4.19	2.59	0.39

W_0 , initial weight; W_t , weight at time-point; W_f , final desiccated weight.

sorption was -13.75%. Endion[®] showed a maximum initial increase in weight after 2 days, followed by progressive weight loss. The sorption was -0.68% at 12 weeks. These samples had contracted after dehydration and could easily be removed from their matrices. The Tubli-Seal EWT[®] samples were tacky to blot dry and stained the tissue. The sorption after 12 weeks was -0.73%.

Solubility

Solubility was calculated from the formula:

$$\frac{(W_0 - W_f)}{W_0} \times 100\%$$

where W_f is the final desiccated weight of the sample. The solubility values for various samples after 12 weeks in solution were: AH Plus[®], 0.32%; Tubli-Seal EWT[®], 1.79%; Endion[®], 8.19%; and Apexit[®], 16.43% (Table 1).

Film thickness

The greatest film thickness recorded was AH Plus[®] (0.44 mm) and the least Tubli-Seal EWT[®] (0.11 mm) (Table 2). The thickness of the AH Plus[®] was double that of any of the other materials, the remaining four having relatively similar values for film thickness. The ranking order for the film thickness of the five cements in increasing order was: Tubli-Seal EWT[®] ≤ Apexit[®] < Endion[®] = Roth 801[®] < AH Plus[®].

Flow

The results of the flow measurements are set out in Table 2. All sealers exhibited similar flow characteristics.

Working and setting time

Table 3 presents the working and setting times for all sealers. The working time values in increasing order were: Endion[®] < Roth 801[®] < Apexit[®] < Tubli-Seal EWT[®] = AH Plus[®]. No change was observed in the AH Plus[®] samples until after 240 min, after which some tackiness was noted. At 300 min, a smooth indentation was left. No marks were left at 500 min, and the setting time for AH Plus[®] was taken to be 8 h and 20 min. The Endion[®] samples became sticky after 30 min.

Separation of Roth 801[®] samples occurred at 31 h with brown fluid on the surface of the samples. The sealer did not set over the 3-month period. The five Roth 801[®] samples mixed on glass slides began to thicken on the 8th day, and this was taken as their setting time.

Discussion

A range of sealer types with differing compositions was selected for evaluation. Calcium hydroxide-containing sealers have been repeatedly associated with favourable healing (Holland & Souza 1985, Tagger & Tagger 1989, Leonardo *et al.* 1997, Holland *et al.* 1998, Tanomaru Filho *et al.* 1998). However, their sealing ability has been questioned (Tagger *et al.* 1988). ZOE sealers have potent antibacterial potential (Fuss *et al.* 1997, Mickel & Wright 1999), and resin cements such as AH Plus[®] are renowned for their low solubility (Grossman 1978, Ørstavik 1983) and adherence to other materials (Grossman 1976, Gettleman *et al.* 1991, Pecora *et al.* 2001). Glass ionomer sealers have been shown to adhere to dentine (Ray & Seltzer 1991, Weiger *et al.* 1995). They are, however, known to be susceptible to dissolution if exposed to solutions before maturation is complete.

Sealer	Mean film thickness (mm) / standard deviation (mm)	Mean time for 0.2 mL to flow through bore (s)	Rate of flow through bore (mL s ⁻¹)
Water		0.58 ^a	0.35
AH Plus [®]	0.44/0.08	5.60	0.04
Apexit [®]	0.12/0.01	5.40	0.04
Endion [®]	0.18/0.04	5.40	0.04
Roth 801 [®]	0.18/0.02	5.20	0.04
Tubli-Seal EWT [®]	0.11/0.02	5.00	0.04

^aThis was calculated from the time taken for 1 mL of water to flow.

Table 2 Mean film thickness and rate of flow of each sealer

Table 3 Working and setting times of sealers

Sealer	Setting time (min) at 37 °C and 100% RH	Working time (min) at 23 °C and 50% RH
AH Plus [®]	500	>120
Apexit [®]	95	90–100
Endion [®]	80	50–60
Roth 801 [®]	Did not set	80–90
Tubli-Seal EWT [®]	70	>120

In the root canal, the sealer may be exposed to tissue fluid and exudate. It is therefore necessary to determine the effects of prolonged exposure of the sealers to fluid. In this case, water was determined to be the most appropriate fluid. With most materials, there are two competitive processes that take place; one is fluid uptake and the other dissolution. The effect produced is dependent on the material type. The results of this study demonstrate that the rate of dissolution of these materials is much greater than their ability to absorb fluid in all cases. This is not unexpected with materials based on an acid–base setting reaction but was more surprising with a resin-based material such as AH Plus[®]. However, the sorption values recorded were lowest for AH Plus[®] (–0.08%), indicating a small overall weight loss; dissolution exceeded water uptake. Endion[®] water uptake exceeded dissolution for the first 11 weeks; however, a sudden weight loss occurred at week 12. This is not an uncommon failing with these materials and would be more likely to occur with a sealer because the powder:liquid ratio is low. This leads to a large part of the material being formed of the matrix, which is subject to degradation in water with respect to time (Crisp *et al.* 1980). This may explain the weight loss in the last week. A small, steady weight loss was observed for Tubli-Seal EWT[®], but that for Apexit[®] was shown to be large. The solubility values for Apexit[®] were approximately 20 times greater than those for Tubli-Seal EWT[®] or Endion[®], and approximately 200 times greater than that for AH Plus[®]. This suggests that there may be a substantial breakdown. Whilst this may be a problem in terms of sealing, there is likely to be a release of calcium and hydroxyl ions. This effect is similar to that noted by Tagger *et al.* (1988).

The weight loss after final desiccation of the material shows the amount of material lost over the test period. The ranking order for weight loss showed Apexit[®] being approximately 50 times more soluble than AH Plus[®] (Apexit[®] > Endion[®] > Tubli-Seal EWT[®] > AH Plus[®]). The findings of this study corroborate those of Grossman (1978), reporting least weight loss for epoxy resin sealers. The recommended solubility value for root-canal sealers

is 3% (British Standard BS 6934 1988). According to the results of this investigation, AH Plus[®] (0.32%) and Tubli-Seal EWT[®] (1.79%) were well within the recommended range. Further work is required to determine the composition of the constituents that have been lost during the test period.

The film thickness values obtained in this study are higher than those achieved with conventional film thickness measurements. The test for film thickness outlined by British Standard BS 6934 (1988) involves using a 15-kg weight, which far exceeds the forces applied clinically. The lower load used in this study (200 g) is more representative of the load applied to root-canal walls during gutta-percha compaction (Blum *et al.* 1997). The values here are not directly comparable with the film thickness of 50 µm set out in the International Standard. The potential importance of the higher film thickness of AH Plus[®] may become more apparent if the need for re-treatment occurs. The combination of greater thickness and lower solubility may make for greater difficulty in its removal.

The ability of sealer to flow and enter uninstrumented accessory root-canal anatomy is important (Gutmann & Witherspoon 1998). The British Standard BS 6934 (1988) defines the flow requirements of a sealer as the production of a 20-mm disc when a 2.5-kg load is applied to 0.5 mL of sealer. The maximum diameters of sealers tested were comparable and ranged between 1.80 and 2.15 mm under a 450-g load. The results of the first flow test (sealer extrusion through bore) also showed all sealers to have similar flow rates. Most endodontic sealers are pseudoplastic (Uhrich *et al.* 1978) so that their viscosity is reduced and flow is increased when shear rate increases during compaction. This should facilitate sealer flow into accessory anatomy, but by the same token pooled sealer near apical foramina may be extruded. Studies on outcome of root-canal treatment indicate that extrusion of root-filling material, including sealers may compromise periapical healing (Harty *et al.* 1970, Heling & Kischinsky 1979, Swartz *et al.* 1983, Sjögren *et al.* 1990, Smith *et al.* 1993). A balance should be struck in selecting a sealer with appropriate flow and manipulation characteristics. The sealer should flow into accessory anatomy and between gutta-percha cones, without increasing the risk of periapical extrusion.

The working and setting times of sealers are dependent on the constituent components, their particle size, and the ambient temperature and relative humidity (Uhrich *et al.* 1978). There is no stipulated standard working time for sealers, but clinical utility demands that it must be long enough to allow placement and adjustment

of root filling if necessary. Two hours has been considered to be optimal (Grossman 1976). In this investigation, initial tests were carried out with an Oscillating Rheometer, which produces a trace of the changing viscosity of the mixed sealer. This was abandoned as two cements, Roth 801[®] and Apexit[®], showed no change in trace amplitude after 24 h. Flow characteristics were then used to determine the working time. It was therefore decided to investigate working time as a function of sealer flow, as previously described. All sealers demonstrated very extended working times. The setting times of sealers obtained in this study were similar to those presented by Grossman (1976).

The British Standard BS 6934 (1988) uses a Gilmore-type indenter to measure the setting time and recommends a value of no more than 72 h at 37 °C and 100% RH. A shortcoming of this indentation method is that the Gilmore needle is hand-held, and standardization of the force applied each time is not possible. The setting times of the sealers in increasing order were: Tubli-Seal EWT[®] < Endion[®] < Apexit[®] < AH Plus[®] < Roth 801[®]. The results recorded ranged from 70 min for Tubli-Seal EWT[®] to 8 h and 20 min for AH Plus[®]. The Roth 801[®] did not set under the test conditions; however, it must be noted that the volume of sealer mixed here is far in excess of that used clinically. When a volume equivalent to that in clinical use was mixed, Roth 801[®] set over 8 days. The sealers investigated in this study demonstrated comparable and acceptable flow rates, working and setting times as set by British Standard BS 6934 (1988).

Conclusions

- 1 AH Plus[®] was found to have the lowest solubility value. The solubilities of Endion[®] and Apexit[®] were high being 8.1 and 16.4%, respectively.
- 2 AH Plus[®] showed the greatest film thickness.
- 3 The flow rate and working time for all sealers were within an acceptable range.
- 4 The setting time for Roth 801[®] exceeded the recommended value under the test conditions.

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